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# TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

U.S. APPLICATION NO. (If known, see 37 CFR 1.51
TBA 10/019967

| INTERNATIONAL APPLICATION | INTERNATIONAL FILING DATE | PRIORITY DATE CLAIMED |
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| PCT/FI00/00397 | 04 May 2000 (04.05.00) | 14 May 1999 (14.05.99) |

TITLE OF INVENTION
A METHOD FOR EXTRACTING COPPER FROM AN AQUEOUS SOLUTION

APPLICANT(S) FOR DO/EO/US
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Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is **FIRST** submission of items concerning a filing under 35 U.S.C. 371.

2. ☐ This is **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.

3. ☒ This express request to begin national examination procedures (35 U.S.C. 371(f) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S. C. 371 (b) and PCT Articles 22 and 39 (1).

4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.

5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))

   a. ☐ is transmitted herewith.
   b. ☒ has been transmitted by the International Bureau.
   c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).

6. ☐ A translation of the International application into English (35 U.S.C. 371(c)(2)). with oath

7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))

   a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☐ have been transmitted by the International Bureau.
   c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   d. ☒ have not been made and will not be made.

8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).

9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). (executed)

10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

**Items 11. to 16. below concern document(s) or information included.**

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.

12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.

13. ☐ A FIRST preliminary amendment.

    ☐ A SECOND or SUBSEQUENT preliminary amendment.

14. ☐ A substitute specification.

15. ☐ A change of power of attorney and/or address letter.

16. ☒ Other items or Information:

- Copy of Notification Informing Applicant of the Communication of the International Application to the Designated Offices
- Copy of International Preliminary Examination Report
- Copy of International Application Published Under the Patent Cooperation Treaty (PCT) No. WO 00/69538
- Check in the amount of $1,124.00
- Return postcard.

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| U.S. APPLICATION NO. (if known, see 37 C.F.R. 1.51) | INTERNATIONAL APPLICATION NO. | ATTORNEY'S DOCKET NO. |
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17. ☒ The following fees are submitted:

| | CALCULATIONS | PTO USE ONLY |
|---|---|---|
| **BASIC NATIONAL FEE** (37 CFR 1.492 (a) (1) - (5) ): | | |
| Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2) paid to USPTO and International Search Report not prepared by the EPO or JPO ..... $1040.00 | | |
| International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO ... $890.00 | | |
| International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2) paid to USPTO ..... $740.00 | | |
| International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33 (1) - (4) ..... $710.00 | | |
| International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1) - (4) ..... $100.00 | | |
| ENTER APPROPRIATE BASIC FEE AMOUNT = | $1,040.00 | |
| Surcharge of **$130** for furnishing the oath or declaration later than ☐20 ☐30 months from the earliest claimed priority date (37 CFR 1.492(e)). | $- - 0.00 | |

| CLAIMS | NUMBER FILED | NUMBER EXTRA | RATE | | |
|---|---|---|---|---|---|
| Total claims | 18-20 | 0 | X $18.00 | $ 0.00 | |
| Independent claims | 4-3 | 1 | X $84.00 | $ 84.00 | |
| MULTIPLE DEPENDENT CLAIM(S) (if applicable) | | | + $280.00 | $ 0.00 | |
| **TOTAL OF ABOVE CALCULATIONS =** | | | | $ 1,124.00 | |
| Reduction of ½ for filing by small entity, if applicable. A Small Entity Statement must also by filed (Note 37 CFR 1.9, 1.27, 1.28). | | | | $ 0.00 | |
| **SUBTOTAL =** | | | | $ 1,124.00 | |
| Processing fee of **$130.00** for furnishing the English translation later than ☐20 ☐30 months from the earliest claimed priority date (37 CFR 1.492(f)). + | | | | $-- 0.00 | |
| **TOTAL NATIONAL FEE =** | | | | $ 1,124.00 | |
| Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). **$40.00** per property = | | | | $ 00.00 | |
| **TOTAL FEES ENCLOSED** | | | | $ 1,124.00 | |
| | | | | **Amount to be refunded:** | $ |
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a. ☒ A check in the amount of $1,124.00 cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. 13-4500 in the amount of to cover the above fees.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 13-4500, ORDER NO. 6009-4616 . A duplicate copy of this sheet is enclosed.

**NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.**

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SCANNED #

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# A METHOD FOR EXTRACTING COPPER FROM AN AQUEOUS SOLUTION

The present invention relates to a method for extracting copper in liquid-liquid solvent extraction from aqueous solutions with a high sulphate content, by raising the viscosity of the extraction solution and by dispersing the aqueous solution into drops, achieving a dense drop aggregation. The viscosity of the extraction solution may be raised either by increasing the content of the actual extractant, the extraction reagent, in the extraction solution or by using a diluting agent with a higher viscosity than that of the diluting agent normally used. By raising the viscosity of the extraction solution the mixing durability of the extraction dispersion can be increased and resulting of that, the amount of residual drops is decreased. Other advantages are that the extraction solution flow of the extraction process decreases in relation to the flow of the aqueous solution acting as the copper source and that the size of the extraction equipment needed is reduced.

Dilute aqueous solutions form when poor copper ores are leached directly. The copper content of such solutions is usually of the order of 1 – 4 g/l Cu. In addition neutral salts often accumulate in the solution, mainly aluminium and magnesium sulphates. Although the copper content does not rise above 1.5 g/l, the sulphate content may rise above 40 g/l, to between 40 and 120 g/l. Some of the sulphate may originate from the ore or the possible use of seawater. In the extraction process the aqueous solution is in a cycle between the extraction and the leaching and thus gradually accumulates the salts which raise the viscosity. Neutral salts can easily cause a viscosity increase harmful to the aqueous solution, even 3 cP, which also disturbs the dispersing of the aqueous and extraction solutions and results in high amounts of residual drops. In particular when dispersion is desired where the organic solution is continuous and the water in drops, an increased viscosity in the aqueous solution can make it difficult to achieve such a dispersion. Previously

the increased viscosity caused by neutral salts and the resulting disadvantages were not taken into account.

In copper extracting processes a mixed organic extraction solution and aqueous solution is generally used in the ratio of O/A (organic/aqueous) 1.0 – 1.2. Present-day copper extracting processes usually follow recommendations given by extractant manufacturers, according to which the organic and aqueous solution of the extraction O/A ratio at all extraction stages of the extraction process should be of the order of 1.0, and the extractant content raised to 3.3 – 4.2 vol.% per every gram of copper, which comes in the direction of flow of the first extraction stage of extraction. In practice this means that if the Cu content of the aqueous solution is 1.5 g/l, the extractant content is maximum 6.3 vol.% according to the recommendations. Generally, when the amount of copper in the solution increases, the amount of extractant decreases relatively. The type of extractant is a chelating copper complexing agent, usually hydroxyoxyme, which forms a strong complex with copper, and one fact affecting the progress of copper extraction is how much extractant is present in relation to the amount of copper to be extracted.

Generally, alifatic or aromatic hydrocarbons, kerosenes, with a distillation range between 190 – 245 °C are used as the diluting agent for the copper extractant. The viscosity of these substances is usually below 2 cP, and for aromatics even below 1.5 cP. It is also possible to us mixtures of aromatic and alifatic hydrocarbons as the diluting agent, where the aromatic content of the mixture is around 20 – 30 vol.%.

As previously stated, in copper extraction it is difficult to get a dispersion of an aqueous solution with a high sulphate content, minimum 40 g/l, where the organic phase is continuous and the aqueous solution in drops, although this is essential in order to improve extraction performance. According to the present invention, the viscosity of the extraction solution is now raised to the

area of 3 –11 cP, and this takes place either by raising the extractant content or by using a diluting agent with a high viscosity in the extraction solution. In addition to this, that the organic phase has been made continuous, the method has proved to have many other advantageous consequences. The essential features of the invention will become apparent in the attached patent claims.

A rise in the viscosity of the extraction solution clearly raises the mixing durability of the extraction dispersion. In this connection a mixing-durable dispersion means a dispersion where no drops below 0.2 mm appear when the mixing intensity is max. 0.15 kWh/m$^3$ in a mixing volume of 50 m$^3$. Volume-specific mixing power is dependent on the mixing volume so that the power required decreases slightly as the volume increases. Obviously the mixing itself also affects mixing durability. The mixers described in US patent 5,185,081 have been settled on to use in the method according to the present invention. These mixers have a double helix, which helps to avoid locally increasing shear rate forces and the small drops generated as a result. When the viscosity of the organic phase has been raised according to the invention and the extraction dispersion made heavier and this dispersion combined with a very smooth, thoroughly uniform mixing of controlled intensity in the mixing area, the conditions are achieved where an evenly distributed mixing energy is not sufficient to attain a turbulence to form droplets. An evenly attenuated mixing creates a dispersion where the drop size is uniform and which thus possesses good separation characteristics. Since the amount of residual drops is small, the extraction result is clearly improved.

In addition to the increase in viscosity of the extraction solution, another key factor is the mixing ratio of the solutions. The denser the drop aggregation, the heavier and simultaneously more mixing-durable the dispersion. The most advantageous result is obtained when a dispersion is formed, where the extraction solution is continuous and the amount of water drops is raised.

When the viscosity of the extraction solution is raised, it has been found that an extraction solution with a higher viscosity is better able to keep a larger amount of the aqueous solution than normal inside it as drops. In the method according to our invention it is possible to lower the O/A ratio to between 0.7 – 1.0 without endangering the continuity of the extraction solution. In practice, this means that the extraction solution flow can be reduced in relation to the copper-containing feed solution (the aqueous solution) by the amount previously described. At the same time the extractant content of the extraction solution is increased to the extent that the mass flow of the extraction solution stays unchanged or increases a little. Thus the viscosity of the extraction solution can be raised successfully.

The factor by which the extractant content is raised compared with the normal recommendation in the method according to the present invention, varies between 1.2 – 5, and is preferably between 1.5 – 3. When very dilute copper-containing feed solutions with max. 2 g/l of copper are being treated, the factor may always rise to 5 i.e. according to our invention the extractant content would then be of the order of 7 – 25 vol.%, preferably 15 – 25 vol. %. When the feed solution copper content is between 2 – 4 g/l, the preferred extractant content is in the range of 15 – 30 vol.%. Generally, however, the extractant content does not increase above a content of 30% by volume. The viscosity of the extraction solution in this case rises to between 3 – 7 cP, which is enough to raise it to a clearly higher level than the viscosity of the aqueous solution. Normally, the aim is to achieve an O/A viscosity ratio of between 1.2 – 3, preferably 1.5 – 2. According to the invention, when extracting dilute copper solutions the extractant content in the extraction solution is presently set in the range of 7 – 30 vol.%, preferably 15 – 30 vol.%.

Regarding aqueous solutions containing over 4 g/l of copper, even an ordinary extractant content in the extraction solution gives a fairly good result. For these solutions, the use of an extractant factor of 1.2 – 2.0 times the

recommendation improves the mixing durability of the dispersion. With the method according to the invention, however, it is possible to raise the extractant content in the extraction solution to 25 – 50 vol.%, when the copper content of the aqueous solution is 4 – 8 g/l and even up to 40 – 70 vol.% if the copper content of the solution is over 8 g/l. The viscosity of the extraction solution can also be raised partly or wholly with the use of a diluting agent. The distillation range and viscosity of the diluting agents generally used was mentioned earlier as being rather low. If other diluting agents are used, this can also raise the viscosity of the extractant. Alifatic hydrocarbon products can be chosen with a distillation range in the range of 220 – 275 °C or 240 – 270 °C, and the viscosity of these substances measured at a temperature of +25 °C is 2.7 or 3.2 cP. If it is desired to use aromatic hydrocarbons, the viscosity of hydrocarbons with a distillation span of 230 - 290 °C is about 3 cP. It is also possible to use mixtures of alifatic and aromatic hydrocarbons.

When treating dilute aqueous solutions containing less than 4 g/l of copper, there is a possibility in our invention of using hydrocarbon compounds that boil at a high boiling range as the diluting agent. The use of a diluting agent to increase viscosity is preferred since the diluting agent is always cheaper than the actual extractant. The proportion of diluting agent in the extraction solution can be between 30 – 93%. It is easier to achieve the required rise in viscosity without the density of the extraction solution increasing significantly with alifatic hydrocarbons. The use of alifatic hydrocarbons is also recommended for reasons of industrial hygiene.

It was mentioned above that when raising the viscosity of the extraction solution it is possible to decrease the external pumping of the extraction solution coming to the extraction stage from outside. If the rise in viscosity takes place with an extraction solution diluting agent, it is not possible to decrease pumping. On the other hand, when viscosity is raised with an extractant, external pumping of the extraction solution can be reduced

significantly compared with the amount of copper-containing aqueous solution being conveyed to the extraction stage. If viscosity is raised both by increasing the extractant content and by using the aforementioned diluting agent, the amount of external pumping decreases in the same degree as the extractant content is increased.

The method according to our invention is described in the attached drawings, where

Figure 1 shows a schematic view of the equipment used in the method of the present invention,

Figure 2 shows a stage calculation of the prior art, where the copper content of the aqueous solution (PLS = pregnant leach solution) coming to the extraction stage is 1.5 g/l and the extractant content of the extraction solution is 5 vol. % Acorga M 5640,

Figure 3 shows a stage calculation according to the present invention, where the copper content of the PLS is 1.5 g/l and the extractant content of the extraction solution is 15 vol. % Acorga M 5640,

Figure 4 shows a stage calculation according to the present invention, where the copper content of the PLS is 1.5 g/l and the extractant content of the extraction solution is 25 vol. % Acorga M 5640,

Figure 5 shows a stage calculation according to the prior art, where the copper content of the PLS is 3.0 g/l and the extractant content of the extraction solution is 8.5 vol. % Acorga M 5640,

Figure 6 shows a stage calculation according to the present invention, where the copper content of the PLS is 3.0 g/l and the extractant content of the extraction solution is 15 vol. % Acorga M 5640,

Figure 7 shows a stage calculation according to the present invention, where the copper content of the PLS is 3.0 g/l and the extractant content of the extraction solution is 25 vol. % Acorga M 5640,

Figure 8 shows a stage calculation according to the present invention, where the copper content of the PLS is 6.5 g/l and the extractant content of the extraction solution is 22 vol. % Acorga M 5640,

Figure 9 shows a stage calculation according to the present invention, where the copper content of the PLS is 6.5 g/l and the extractant content of the extraction solution is 30 vol. % Acorga M 5640,

Figure 10 shows a stage calculation according to the present invention, where the copper content of the PLS is 6.5 g/l and the extractant content of the extraction solution is 40 vol. % Acorga M 5640,

Figure 11 shows a stage calculation according to the present invention, where the copper content of the PLS is 2.5 g/l and the extractant content of the extraction solution is 40 vol. % LIX 984N, and

Figure 12 shows a stage calculation according to the present invention, where the copper content of the PLS is 32 g/l and the extractant content of the extraction solution is 50 vol. % Acorga M 5640.

Figure 1 describes a copper extraction process for treating dilute copper solutions. The process consists of two extraction stages, E1 and E2, one extraction solution washing stage W and one extraction solution stripping stage S. Both the extraction stages and the washing and stripping stages consist of a mixing section 1, a settler 2, and a pump 3 used to transfer the dispersion. The mixing section has at least one mixer, which is preferably equipped with the mixing devices described earlier. The principles of the extraction stages are the types described in e.g. WO patent application publications 97/40899, 97/40900, 97/40901 and 97/41938.

As usual, the extraction functions on a counterflow principle, whereby aqueous solution 4 comes first to extraction stage E1 and extraction solution 5 to stage E2. The aqueous solution exiting the final extraction stage E2, raffinate 6, is fed back to ore leaching, and copper-enriched extraction solution 7 is fed from E1 to washing W and stripping S. In practice, the

extraction solution is circulated via storage tanks. Lean electrolyte 8 is fed from electrolysis to the stripping stage where the copper contained in the organic phase is extracted. The aqueous solution 9 containing copper sulphate exiting the stage goes as rich electrolyte to electrowinning and the stripped organic phase 5 is circulated back to extraction stage E2.

Figure 1 shows how considerably the size of the washing and stripping stages of the extraction process is reduced when an extractant is used according to the invention to raise the viscosity of the extraction solution. In fact the reduction is in direct ratio to the external extraction solution pumping, because the mixer-settlers in question are dimensioned directly with the solution flows in all respects, pumping, mixing and solution separation.

Therefore, in cases where the extractant content is raised for example to double the amount normally used, and external extraction solution pumping is correspondingly decreased to half the normal flow, the mixer and settler volumes of the washing and stripping stages are halved. The actual extraction stages E1 and E2 remain almost their earlier size and the same external extraction solution pumping goes through them, but the extraction solution can be circulated within the stages in order to maintain extraction solution continuity. The extraction solutions flow through each stage of the extraction equipment at essentially the same time. As mentioned above, the O/A mixing ratio of the solutions may be reduced according to the method of the invention to below 1 to a value between 0.7 – 1.0, and the size of the extraction stages equipment can be reduced correspondingly.

When estimating the effect of our invention on the size of the extraction equipment, it should be noted that Fig. 1 is only indicative of the relative size of the extraction stages and that of the washing and stripping stages. There are often two stripping stages in an extraction plant and in some cases also two washing stages. Then the savings made by reducing the size of the

equipment are correspondingly greater. The amount of extraction solution inside the extraction plant is also reduced correspondingly even if the changes in the amount of extractant itself are not large, since the content of extractant in the extraction solution has been raised. In certain cases it is expedient even to raise the amount of extraction agent circulating in the process, so that the advantages described in the method can be achieved in full measure.

The method according to our invention provides the opportunity to treat difficult impurities such as copper ore containing chloride, nitrate or manganese in an economical way. In particular, ores containing a lot of iron are generally problematic, because iron increases the transfer of the above-mentioned impurities to the electrolyte via the extraction solution. This results in a situation where it is even more important than before to prevent the transfer of said impurities first to the extraction solution with the unseparated drops of aqueous solution from extraction stage E1 to washing stage W and from there on to stripping stage S.

According to our invention it is now possible to use equipment that is smaller than usual in the washing stage, but as it is known on the other hand, a prolonged settling time in the washing stage (larger settler) improves the separation of impurities. Now it is possible to enlarge the washing stage, in particular its settler section in relation to the extraction solution flow used, for instance the size of a settler according to the conventional method without increasing costs and to achieve better separation of impurities than before. In practice this means that in the washing and stripping stages the mixing and separating times are longer, i.e. the solutions flow through them more slowly than through the actual extraction stages. With this system our method offers the possibility for flexible, case-specific dimensioning.

When poor copper ores are processed by direct leaching, the result, as stated above, is an aqueous solution with a copper content in the range of 1 – 4 g/l, and in addition neutral salts accumulate in the solution. The sulphate content of the solution may rise to between 40 – 120 g/l, which causes a rise in the viscosity of the aqueous solution, but on the other hand, sulphate has the benefit of acting as a pH buffer when using copper extraction in the pH range of 0.8 – 2.2. In other words it improves the equilibrium of the copper extraction and makes more copper transfer to the extraction solution.

Example 1

A series of tests were carried out, which show that the separation of the organic solution and the aqueous solution from each other improves when the viscosity of the organic phase is raised by increasing the extractant content in copper extraction according to our invention. Table 1 presents the composition of the extraction solution and the results obtained.

The aqueous solution was made using ion-exchanged water, copper sulphate and sulphuric acid. The copper content of the solution was 2 g/l, the sulphate content 52 g/l and the pH 1.8. The extraction solution was prepared by mixing the commercial extractants shown in the table in different proportions with a commercial kerosene solution D70 as diluting agent. Mixing contact was made between the extraction solutions and the copper sulphate solution (aqueous solution) at room temperature and in the O/A phase ratio of 1.0, thereby obtaining the copper content values of the solutions in the table. After mixing all the solutions were recovered and stored for two weeks before the actual mixing tests. This ensured that the extraction solutions in particular corresponded to the solutions used in normal extraction, without the drawbacks of new extraction solutions.

A double helix agitator as described in US patent 5,185,081, with a diameter of 152 mm and height of 174 mm was used in the mixing tests. The mixer itself

was a flat-bottomed cylinder with a diameter of 214 mm and effective solution depth also of 214 mm. The cylinder was equipped with four baffles positioned on the frame of the cylinder, with a width of 18 mm and at a distance of 3.5 mm from the inner surface of the cylinder.

The mixing contact itself was made at room temperature and in the O/A phase ratio of 1.0 so that the extraction solution was continuous in all tests and the aqueous solution in drops. The revolution speed of the mixer was 220 rpm and the duration of mixing was 3 minutes in all tests. In all tests each extraction solution was mixed with a new batch of the aqueous solution. After mixing the solutions were made to separate by the effect of gravity. 15 minutes after mixing the amount of residue solution in each of the separated solutions was determined. The drop residues (entrainment levels) are shown in Table 1, where A/O means water in the extraction solution and O/A means organic phase drops in the aqueous solution.

Table 1

| Test | Extr.sol. | | | | Viscos. | Cu/extr.sol. | Residual drops | |
|---|---|---|---|---|---|---|---|---|
| | Extractant | | Dil. agent | | | | A/O | O/A |
| | | til.-% | | til.-% | cP | g/l | ppm | ppm |
| 1 | Acorga M5640 | 5 | D70 | 95 | 2,7 | 2 | 500 | 90 |
| 2 | Acorga M5640 | 8,5 | D70 | 91,5 | 3 | 3,5 | 450 | 70 |
| 3 | Acorga M5640 | 15 | D70 | 85 | 3,3 | 6,2 | 150 | 40 |
| 4 | Acorga M5640 | 25 | D70 | 75 | 4,2 | 7,4 | 100 | 25 |
| 5 | Acorga M5640 | 30 | D70 | 70 | 4,9 | 13,7 | 150 | 25 |
| 6 | LIX984N | 40 | D70 | 60 | 6,1 | 19,8 | 50 | 15 |
| 7 | Acorga M5640 | 50 | D70 | 50 | 8,2 | 20,1 | 50 | 12 |

The tests show that raising viscosity by increasing the extractant (extraction reagent) content clearly decreases the amount of residual drops in the settled solutions.

Example 2

An aqueous solution was prepared with a copper content of 1.5 g/l, sulphate content of 50 g/l and pH of 1.8. Three different extraction solutions were also prepared:

| | | |
|---|---|---|
| 1. | Acorga M5640 | 5.0 vol.-% |
| | D70 | 95 vol.-% |
| 2. | Acorga M5640 | 15.0 vol.-% |
| | D70 | 85 vol.-% |
| 3. | Acorga M5640 | 25.0 vol.-% |
| | D70 | 75 vol.-% |

The first solution represents an extraction solution according to the prior art.

Extraction equilibrium curves EEQ and stripping equilibrium curves SEQ shown in Figs 2, 3 and 4 were defined for the extraction solutions and aqueous solutions in question with the method used by experts in this field. Diagram 2 shows the prior art, diagrams 3 and 4 the method according to this invention. Next, making use of the equilibrium data in question, an extraction calculation was made for a copper extraction process with two extraction stages functioning on the counterflow principle and two stripping stages. The calculation was made on the basis of the McCabe-Thiele method, familiar to specialists in the field. The extraction and stripping stages reach as far as the equilibrium curve, because the stage efficiency is very high when using for instance the equipment described in WO patent publications.

The three stage calculations presented in the example show that the copper extraction yield remains at a good level and almost unchanged, even though the external pumping of the extraction solution is reduced. The copper contents of the raffinate are in all cases 0.2 – 0.4 g/l. The lowest content is obtained by raising the extractant content to 15%, whereby the external pumping of the extraction solution can be reduced to 35% of the PLS

(pregnant leach solution) feed i.e. copper-containing aqueous solution feed. It is apparent from the stage calculation in question that the extraction equilibrium remains good in extraction conditions (the EEQ curve rising steeply when the Cu content of the aqueous solution is under 0.5 g/l) over its extraction solution copper content level, set for the extraction solution after the second stripping stage S2 (dotted line BO, e.g. 3.0 g/l in Figure 3).

Another essential factor is that the stripping equilibrium allows the making of strong copper electrolyte when the copper content of the extraction solution can be lowered sufficiently.This means the level where the extraction equilibrium is still rising sharply in an aqueous solution Cu content of under 0.5 g/l, as stated earlier. The stage calculation reveals that with the method according to the present invention, a significant improvement is achieved in the copper content of the electrolyte going to copper electrolysis. With two stripping stages the electrolyte was made almost saturated as regards copper sulphate.

In different stage calculations the copper electrolyte developed as follows: in a normal copper process (extractant content 5 vol.%) the copper content of the "poor" electrolyte (LEL = lean electrolyte) coming to the washing stage is to be kept as low as 34 g/l and in the "rich" electrolyte (REL = rich electrolyte) the content may rise to the value of 42.5 g/l. In the present method the corresponding values are 36 g/l and 50 g/l when using an extractant content of 15 vol. % and 36 g/l and 51.5 g/l when using an extractant content of 25 vol. %.

In the method according to the present invention a smaller amount than usual of extraction solution is circulated in relation to the PLS solution. Likewise the electrolyte circulation is correspondingly much smaller. In order to describe the information included in the stage calculations the circulation of the solutions can be checked for instance on the basis of diagram 3. The

stage calculation is in two parts: extraction on the left and stripping on the right. The solution compositions of the different stages can be seen at the intersection points of the stages and the equilibrium curves. For example, in extraction stage E1 the Cu content of the aqueous solution decreases from the PLS content of 1.5 g/l to 0.6 g/l and the Cu content of the extraction solution rises from the E2 content of 4.2 g/l to 6.7 g/l. In stripping stage S1 the extraction solution on the other hand falls from an LO (loaded organic) value of 6.7 g/l to 4.2 as the Cu content of the copper electrolyte rises from 40.4 g/l to 50.0 g/l. The Cu content of the extraction solution falls further to a BO (barren organic) value of 3.0 in stripping stage S2, from where the extraction solution moves on to extraction stage E2 of the extraction.

The important points in said stage calculations are the so-called operating lines which indicate the contents in which the solutions touch each other when entering or leaving the first extraction and stripping stages E1 and S1 and the final extraction and stripping stages E2 and S2 and in between said stages. From the nature of the stage calculations it follows that the gradient of the operating lines indicate the external solution pumping i.e. the ratio of PLS and extraction solution flows in extraction as well as the ratio of electrolyte and extraction solution flows in stripping. It has been possible to calculate from the external pumping ratios how much the electrolyte circulation is reduced with our method in relation to the amount of PLS flow.

Certain figures characteristic of our invention have been assembled in Table 2, when the extractant contents of the extraction solution are 15 and 25 vol.% and these figures are compared with figures for conventional copper extraction, where the extractant content is 5 vol. %. The copper content of the aqueous solution (PLS) is 1.5 g/l in all cases. The extractant is Acorga M5640 and the diluting agent kerosene D70. The temperature of the extraction solution is 18 °C.

Table 2

| Quantity | Kuva 2 | Kuva 3 | Kuva 4 |
|---|---|---|---|
| Extractant content, vol-% | 5 | 15 | 25 |
| Viscosity of extraction solution, cP | 2,7 | 3,3 | 4,2 |
| Cu content of raffinate, g/l | 0,3 | 0,2 | 0,4 |
| Rise in Cu content of extraction solution, g/l | 1 | 3,7 | 4,9 |
| Rise in Cu content of electrolyte, g/l | 8,5 | 14 | 15,5 |
| Rich electrolyte content, g/l | 42,5 | 50 | 51,5 |
| Ext. solution pumping ratio in extraction O/A | 1,2 | 0,35 | 0,24 |
| Ext. solution pumping ration in stripping O/A | 8,5 | 3,8 | 3,2 |
| Flow ratio of electrolyte and PLS | 0,14 | 0,09 | 0,08 |

From this table the advantages given by our invention are apparent. In addition to the raised viscosity of the extraction solution and the fact that the solutions can be separated cleanly, it has been possible to reduce essentially the size of the equipment for handling the extraction solution in the extraction process, such as washing and stripping equipments, as well as other extraction solution equipment for the external circulation of the extraction solution, such as storage tanks and post-separators for separating the residue solutions. Likewise the size of the equipment for handling the electrolyte is reduced, such as flotation and pressure filtration apparatus, any possible post-separators and storage tanks. Another important factor is that the copper content can be raised, ensuring the quality of the copper.

Example 3

In this example the performance values according to the prior art are shown in diagram 5 and the values of the method according to the present invention in diagrams 6 and 7, based on the presented stage calculations. These again show that the method helps reduce the size of the equipment considerably. The copper content of the aqueous solution is 3 g/l, i.e. still a dilute solution. The extractant content of an ordinary solution, given first in the table, is 8.5 vol.% and the following 15 and 25 vol.% according to this invention, as in the previous example. The extractant and diluting agent used are the same as in example 2. The temperature of the extraction solution is 18 °C.

Table 3

| Quantity | Kuva 5 | Kuva 6 | Kuva 7 |
|---|---|---|---|
| Extractant content, vol-% | 8,5 | 15 | 25 |
| Viscosity of extraction solution, cP | 3 | 3,3 | 4,2 |
| Cu content of raffinate, g/l | 0,3 | 0,25 | 0,35 |
| Rise in Cu content of extraction solution, g/l | 2,4 | 4,5 | 6,3 |
| Rise in Cu content of electrolyte, g/l | 9,5 | 14 | 15,5 |
| Rich electrolyte content, g/l | 45,5 | 50 | 51,5 |
| Ext. solution pumping ratio in extraction O/A | 1,13 | 0,61 | 0,42 |
| Ext. solution pumping ration in stripping O/A | 3,96 | 3,11 | 2,46 |
| Flow ratio of electrolyte and PLS | 0,29 | 0,2 | 0,17 |

Example 4

In this example, an aqueous solution was used with a Cu content of 6.5 g/l, in other words richer than is usually achieved with direct leaching of any poor ore. However, even when treating this kind of solution our method has distinct advantages. In the next table the stage calculations of diagrams 8, 9 and 10 were used. The contents of the extraction solution are 22, 30 and 40 vol.%, the extractant Acorga M6540 and the diluting agent again kerosene D70. The temperature of the extraction solution was 18 °C.

Table 4

| Quantity | Fig. 8 | Fig. 9 | Fig. 10 |
|---|---|---|---|
| Extractant content, vol-% | 22 | 30 | 40 |
| Viscosity of extraction solution, cP | 3,7 | 4,9 | 6,3 |
| Cu content of raffinate, g/l | 0,2 | 0,3 | 0,4 |
| Rise in Cu content of extraction solution, g/l | 5,9 | 9,2 | 14,5 |
| Rise in Cu content of electrolyte, g/l | 16 | 16 | 16 |
| Rich electrolyte content, g/l | 52 | 52 | 52 |
| Ext. solution pumping ratio in extraction O/A | 1,07 | 0,67 | 0,42 |
| Ext. solution pumping ration in stripping O/A | 2,71 | 1,74 | 1,1 |
| Flow ratio of electrolyte and PLS | 0,4 | 0,39 | 0,38 |

Example 5

This example studied the possibility of reducing the external pumping of the extraction solution considerably by raising its extractant content to a significantly high level. When the pH of the base solution is close to 2 and the

same aqueous solution contains sulphates, the extraction equilibrium is very beneficial when the copper content of the aqueous solution is low. In the case of the example the Cu content of the aqueous solution is 2.5 g/l, the pH is 1.8 and the amount of sulphates 50 g/l. According to this example the method of the present invention increases the copper extraction yield. As shown by the stage calculation in Fig.11, a very low raffinate content of 0.15 g/l copper is obtained while the external pumping of the extraction solution ratio drops to as low as 0.15. The external pumping of the electrolyte in ratio to the external pumping of the PLS also settles at the same value of 0.15. In this example the extractant used was the commercial chemical LIX 984N, which is similar to the reagent used in the previous example, and again kerosene D70 was the diluting agent. The extractant content was 50 vol.%.

Table 5

| Quantity | Fig. 11 |
|---|---|
| | |
| Viscosity of extraction solution, cP | 8 |
| Cu content of raffinate, g/l | 0,15 |
| Rise in Cu content of extraction solution, g/l | 15,7 |
| Rise in Cu content of electrolyte, g/l | 16 |
| Rich electrolyte content, g/l | 52 |
| Ext. solution pumping ratio in extraction O/A | 0,15 |
| Ext. solution pumping ration in stripping O/A | 1,02 |
| Flow ratio of electrolyte and PLS | 0,15 |

Example 6

The example shows how high the copper content of the PLS can be raised using our invention. In this example the Cu content of the PLS was raised up to 32 g/l while the extractant content of the extraction solution was raised to 50 vol.%, with Acorga M5640 as reagent and kerosene D70 as diluting agent. Mixing with the double helix mixer mentioned above is successful, even though an extractant content of as much as 40 – 70 vol.% is used. It is advantageous to do this when it is desired to reduce external pumping of the extraction solution. The stage calculation concerning 50 vol.% in Fig.12 and the summary in Table 6 clarify these possibilities further.

Table 6

| Quantity | Fig. 12 |
|---|---|
| | |
| Viscosity of extraction solution, cP | 8,2 |
| Cu content of raffinate, g/l | 5 |
| Rise in Cu content of extraction solution, g/l | 13 |
| Rise in Cu content of electrolyte, g/l | 16,5 |
| Rich electrolyte content, g/l | 50,5 |
| Ext. solution pumping ratio in extraction O/A | 2,07 |
| Ext. solution pumping ration in stripping O/A | 1,27 |
| Flow ratio of electrolyte and PLS | 1,63 |

5

PATENT CLAIMS

1. A method for extraction of copper from an aqueous solution containing a large amount of sulphates in liquid-liquid extraction, **characterized in** that the viscosity of an extraction solution is adjusted within the range of 3 –11 cP and that the volumetric ratio of the extraction solution and an aqueous solution in an extraction mixture to between 0.7 – 1.0, whereby the aqueous solution is dispersed into drops in the extraction solution.

2. A method according to claim 1, **characterized in** that the viscosity of the extraction solution is raised by raising the content of an extractant.

3. A method according to claim 2, **characterized in** that the viscosity of the extraction solution is raised by regulating the extractant content of the extraction solution in the range of 15 – 70 vol. %.

4. A method according to claim 1, **characterized in** that the ratio (O/A) between the organic solution and the aqueous solution coming to the extraction stage from outside is regulated in the range of 0.15 – 1.

5. A method according to claim 3, **characterized in** that in treating an aqueous solutions with a copper content of maximum 2 g/l, the viscosity of the extraction solution is raised by adjusting the content of the extractant in the extraction solution to the range of 15 – 25 vol.%.

6. A method according to claim 5, **characterized in** that the external pumping ratio of the extraction solution and the aqueous solution is adjusted to the range of 0.2 – 0.5 and the corresponding external pumping ratio between a stripped copper electrolyte and the aqueous solution of the extraction is adjusted to the range of 0.08 – 0.02.

7. A method according to claim 3, **characterized in** that in treating an aqueous solution with a copper content of 2 – 4 g/l, the viscosity of the extraction solution is raised by adjusting the content of the extractant in the extraction solution to the range of 15 – 30 vol.%.

8. A method according to claim 7, **characterized in** that the external pumping ratio of the extraction solution and the aqueous solution is adjusted to the range of 0.3 – 0.7 and the corresponding external pumping ratio between the stripped copper electrolyte and the aqueous solution of the extraction is adjusted to the range of 0.15 – 0.25.

9. A method according to claim 3, **characterized in** that in treating an aqueous solution with a copper content of 4 – 8 g/l, the viscosity of the extraction solution is raised by adjusting the content of the extractant in the extraction solution to the range of 25 – 50 vol.%.

10. A method according to claim 9, **characterized in** that the external pumping ratio of the extraction solution and the aqueous solution is adjusted to the range of 0.4 – 0.8 and the corresponding external pumping ratio between the stripped copper electrolyte and the aqueous solution of the extraction is adjusted to the range of 0.25 – 0.50.

11. A method according to claim 3, **characterized in** that in treating an aqueous solution with a copper content of over 8 g/l, the viscosity of the extraction solution is raised by adjusting the content of the extractant in the extraction solution to the range of 40 – 70 vol.%.

12. A method according to claim 11, **characterized in** that the external pumping ratio of the extraction solution and the aqueous solution is adjusted within the range of 1 – 4 and the corresponding external pumping ratio between the stripped copper electrolyte and the aqueous solution of

the extraction is adjusted within the range of 0.8 – 3.

13. A method according to claim 1, **characterized in** that the viscosity of the extraction solution is raised by using alifatic hydrocarbons, kerosenes, with a viscosity of 2.7 – 3.2 cP when measured at ambient temperature, as diluting agent for the extraction solution.

14. A method according to claim 1, **characterized in** that the viscosity of the extraction solution is raised by using aromatic hydrocarbons, kerosenes, with a viscosity of about 3 cP when measured at ambient temperature, as diluting agent for the extraction solution.

15. A method according to any of the above claim, **characterized in** that the viscosity of the extraction solution is raised by using a mixture of alifatic and aromatic hydrocarbons, with a viscosity of minimum 2.7 cP when measured at ambient temperature, as diluting agent for the extraction solution.

16. A method according to claim 1, **characterized in** that the sulphate content of the aqueous solution fed to solvent extraction is minimum 40g/l.

17. A method according to any of the above claim, **characterized in** that the extracting solutions flow through each stage of the extraction equipment at essentially the same time.

18. A method according to any of the above claim, **characterized in** that the extracting solutions flow through the washing and stripping stages of the equipment more slowly than the actual extracting stages.

## ABSTRACT

The invention relates to a method for extracting copper in liquid-liquid solvent extraction from aqueous solutions with a high sulphate content, by raising the viscosity of the extraction solution and by dispersing the aqueous solution into drops, achieving a dense drop aggregation. The viscosity of the extraction solution may be raised either by increasing the content of the actual extractant, the extraction reagent, in the extraction solution or by using a diluting agent with a higher viscosity than that of the diluting agent normally used. By raising the viscosity of the extraction solution the mixing durability of the extraction dispersion can be increased and resulting of that the amount of residual drops is decreased. Other advantages are that the extraction solution flow of the extraction process decreases in relation to the flow of the aqueous solution acting as the copper source and that the size of the extraction equipment needed is reduced.

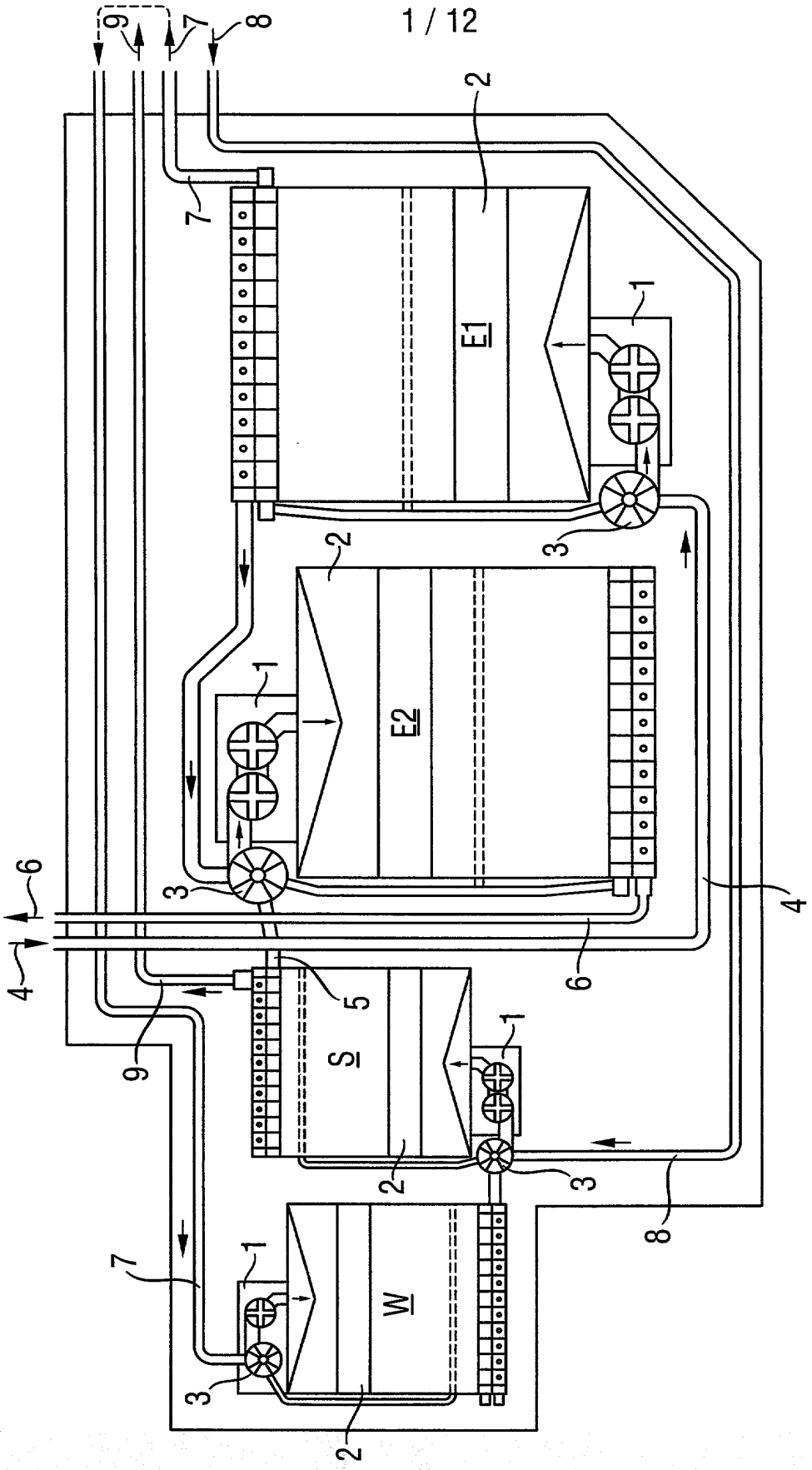


Fig. 1

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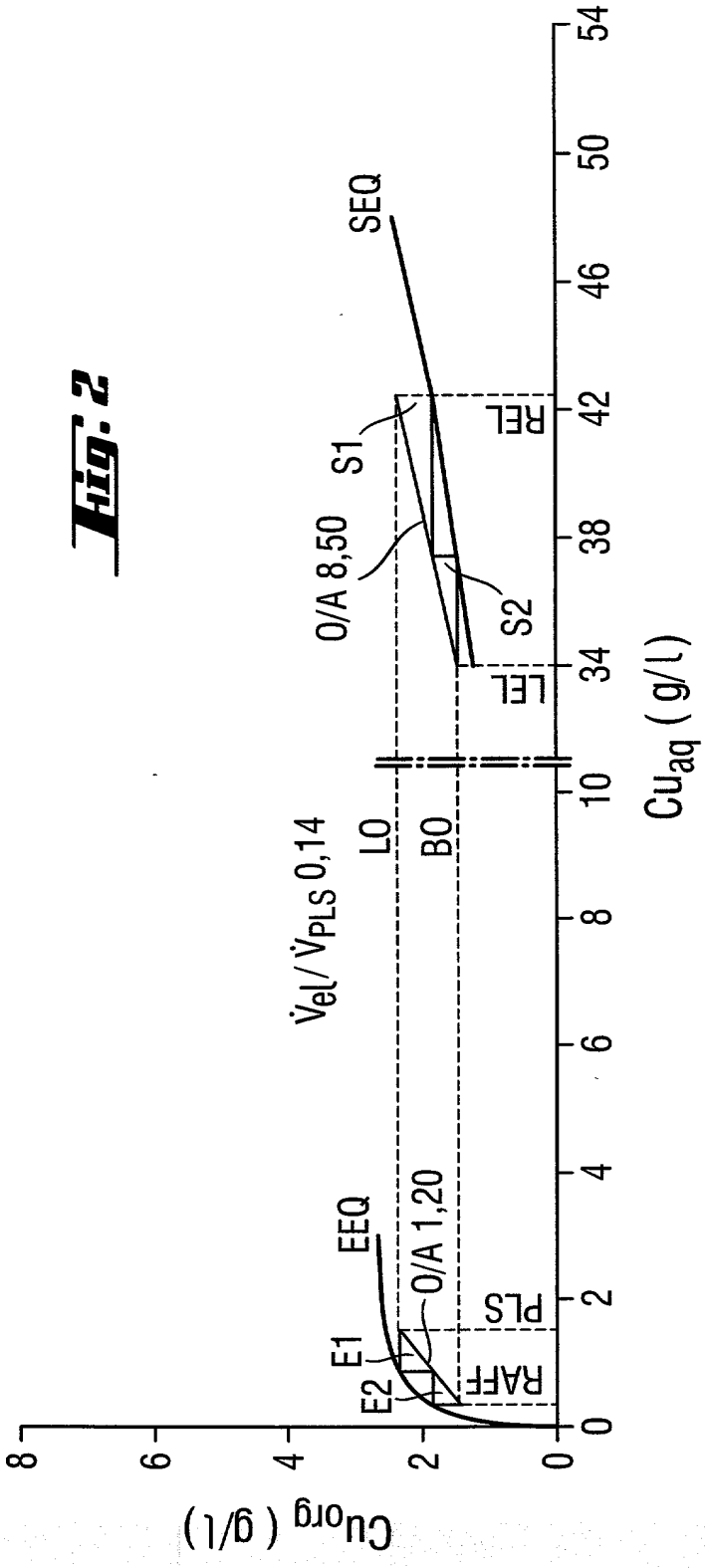
Fig. 2



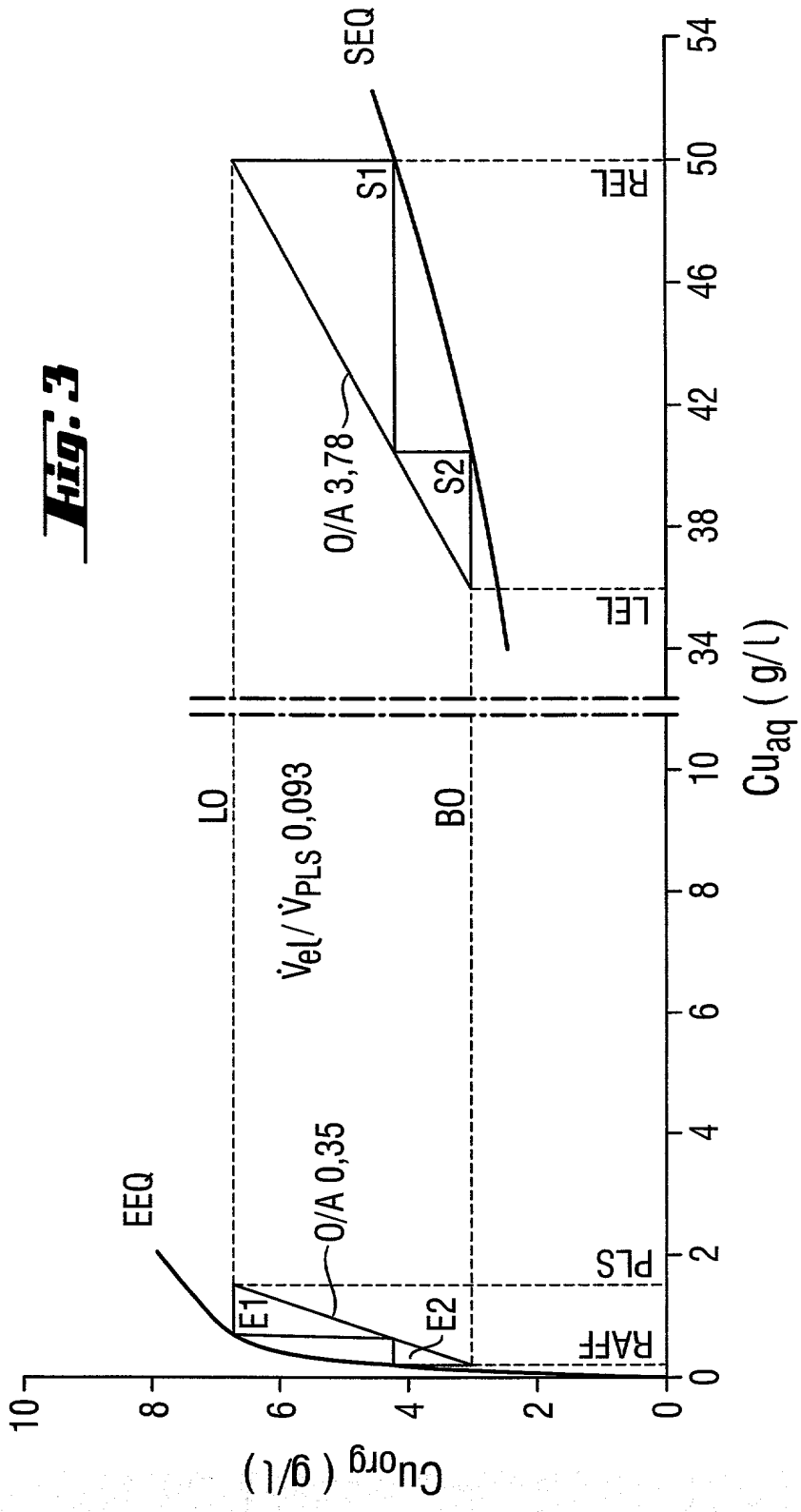

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Fig. 3

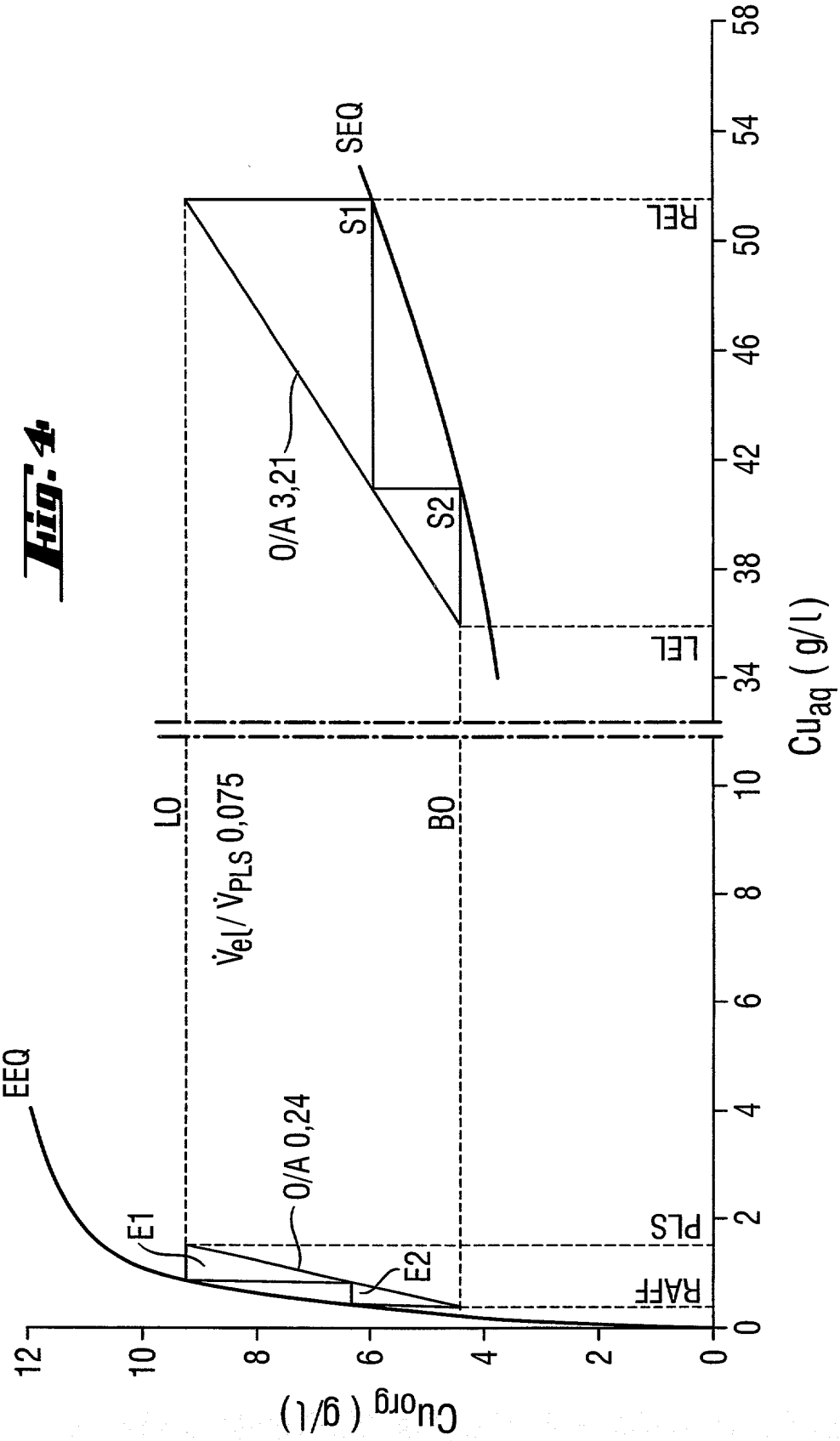
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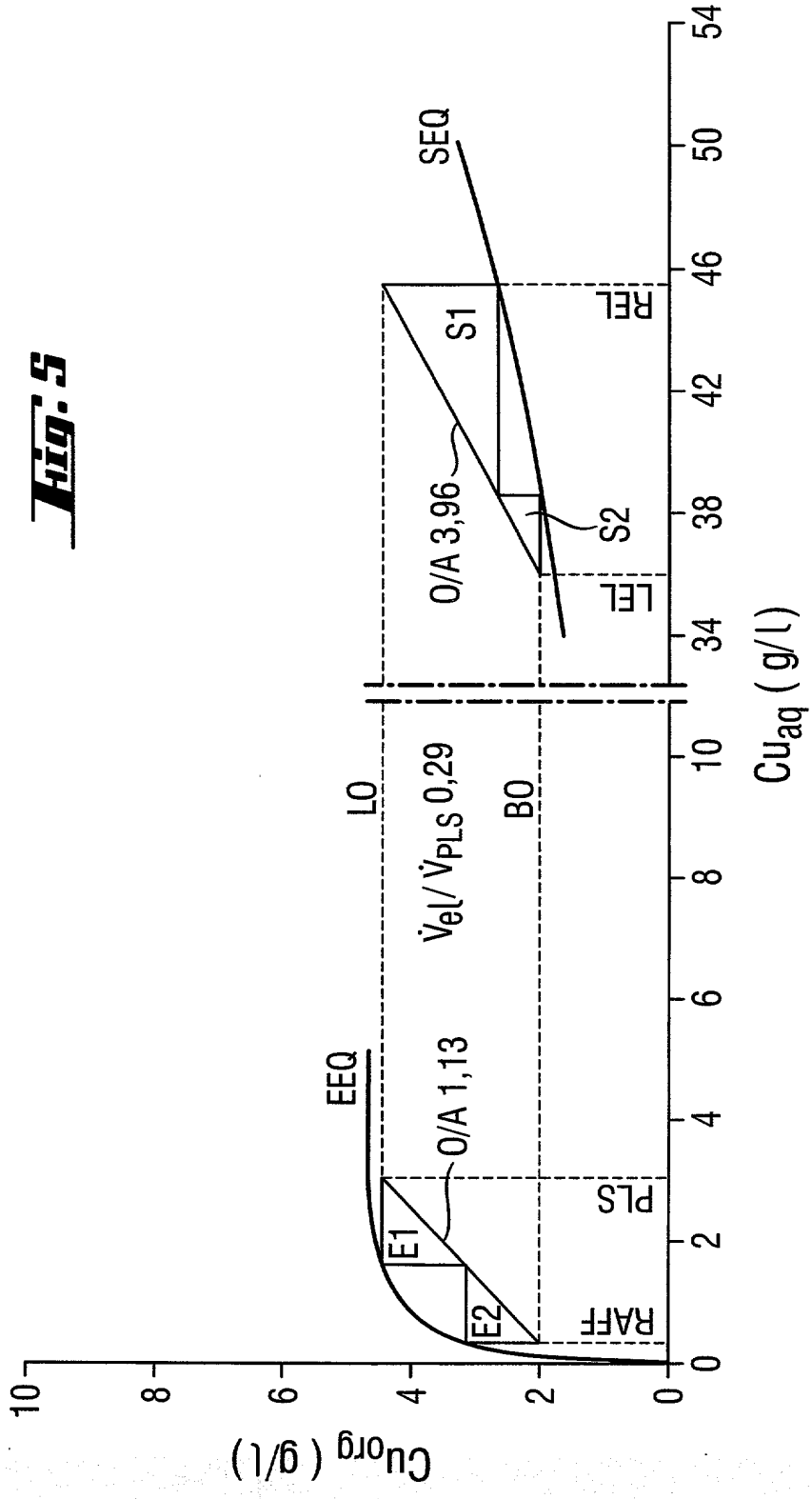
Fig. 4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O/A 3,96
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Fig. 5

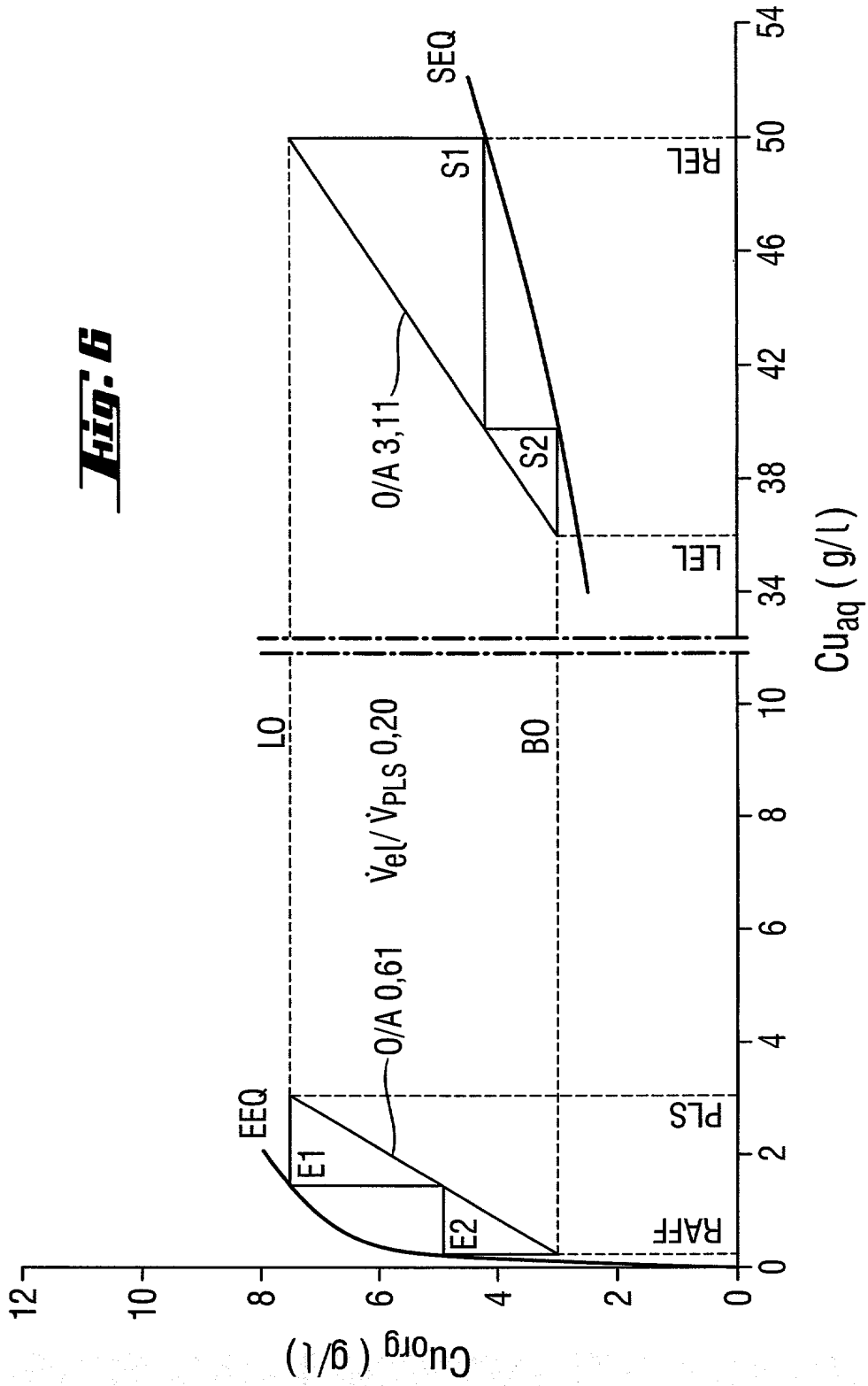
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Fig. 6

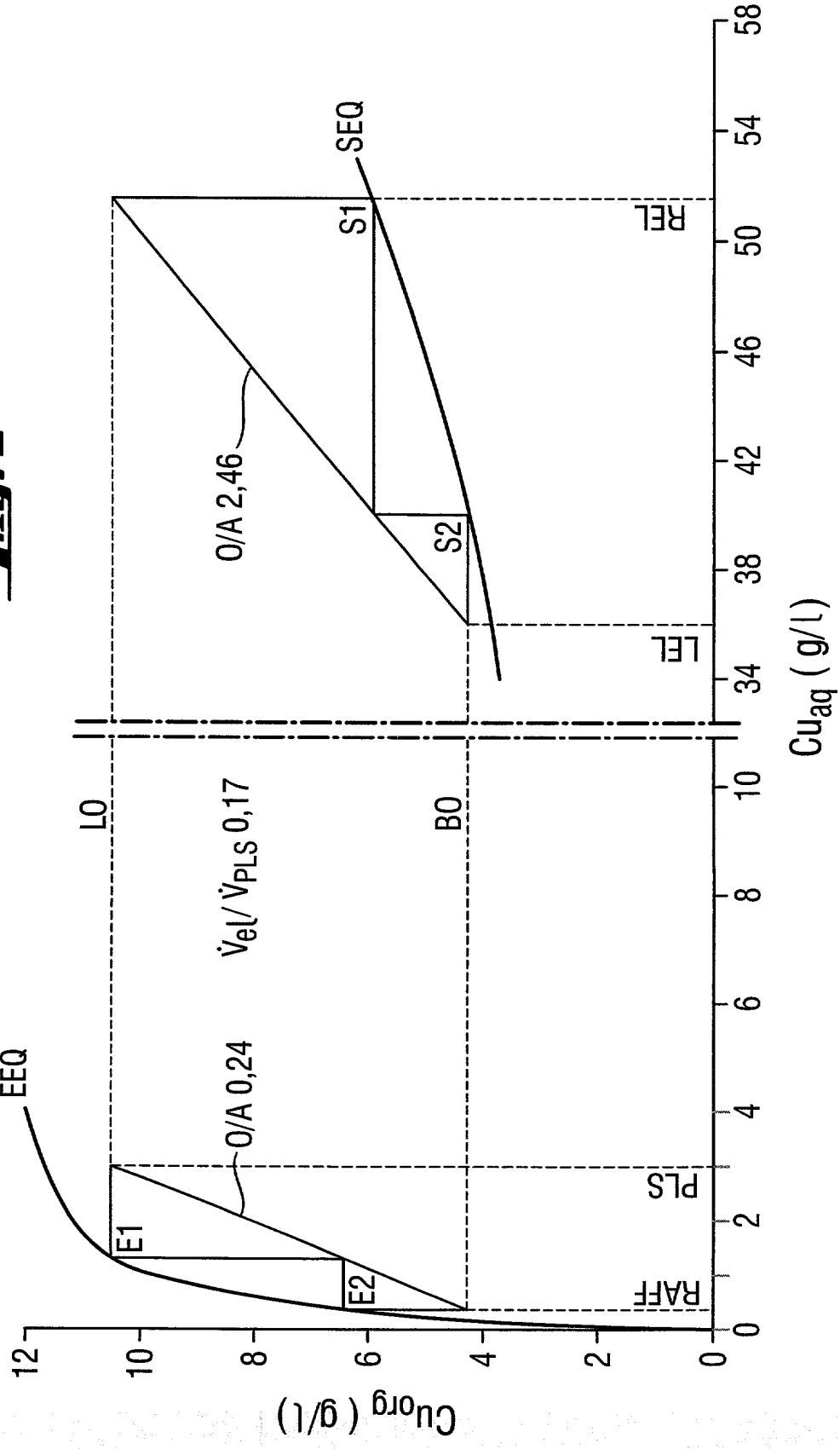
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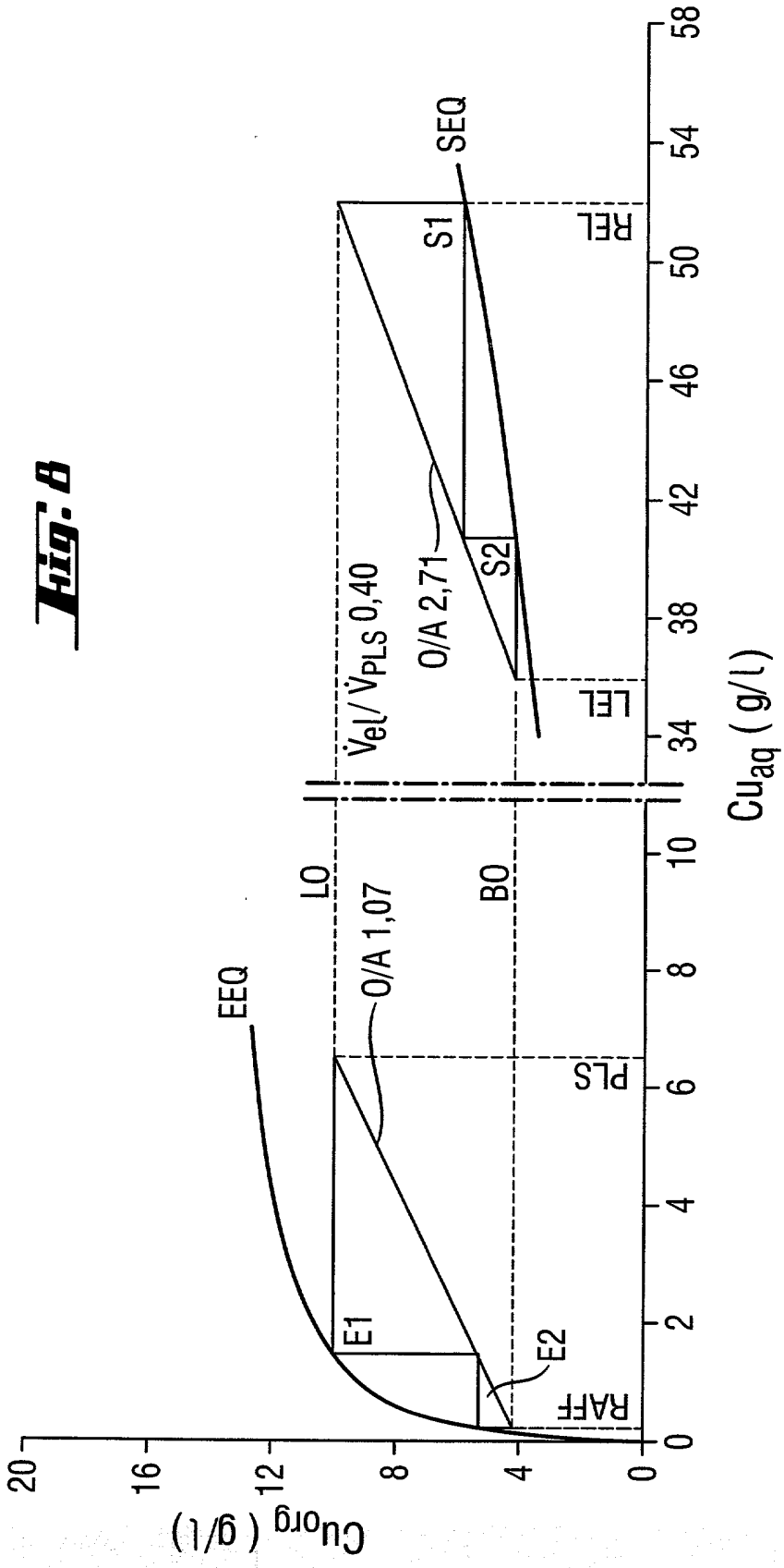
Fig. 1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S2
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LEL

Fig. 8

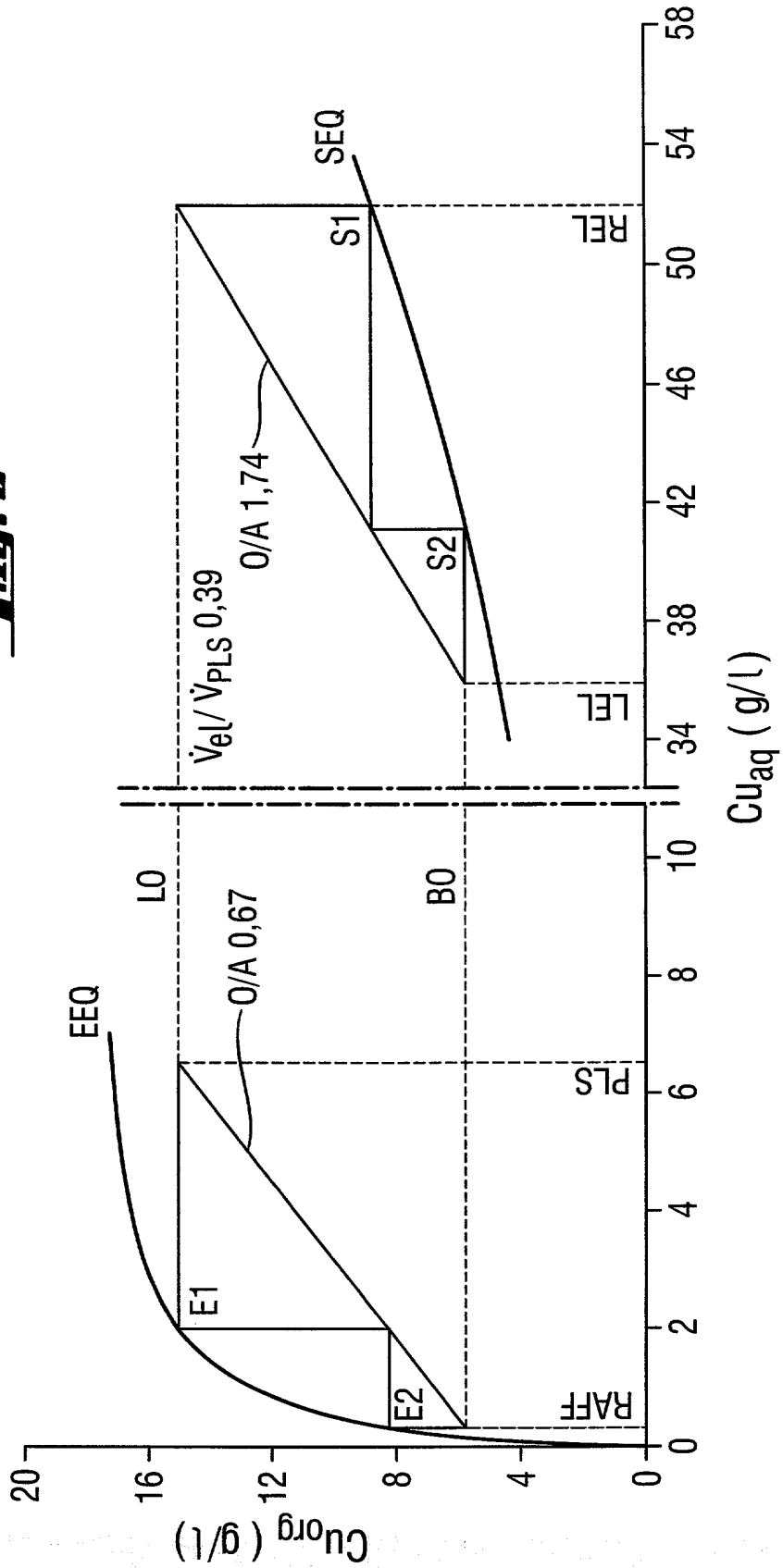
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Fig. 9

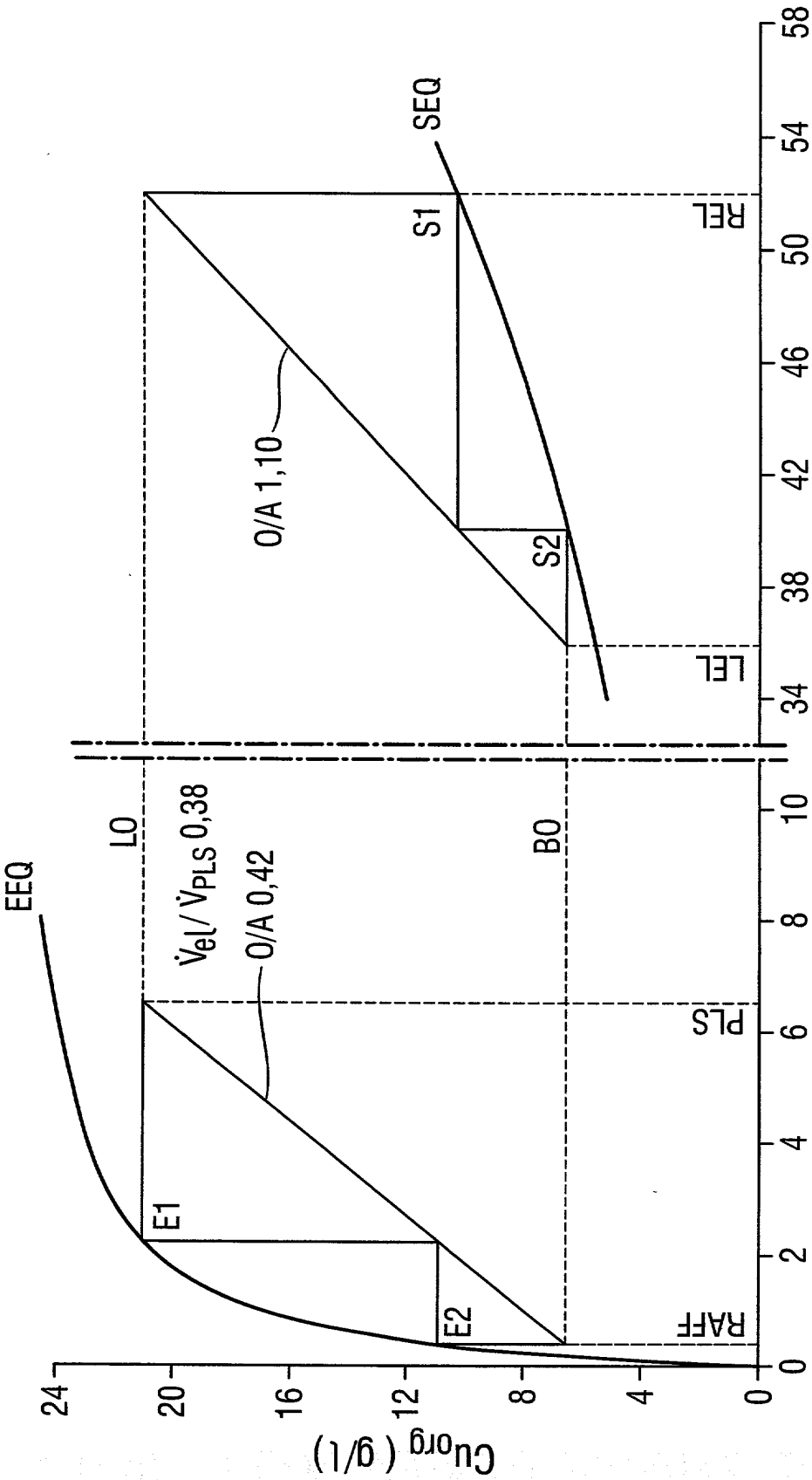
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Fig. 10

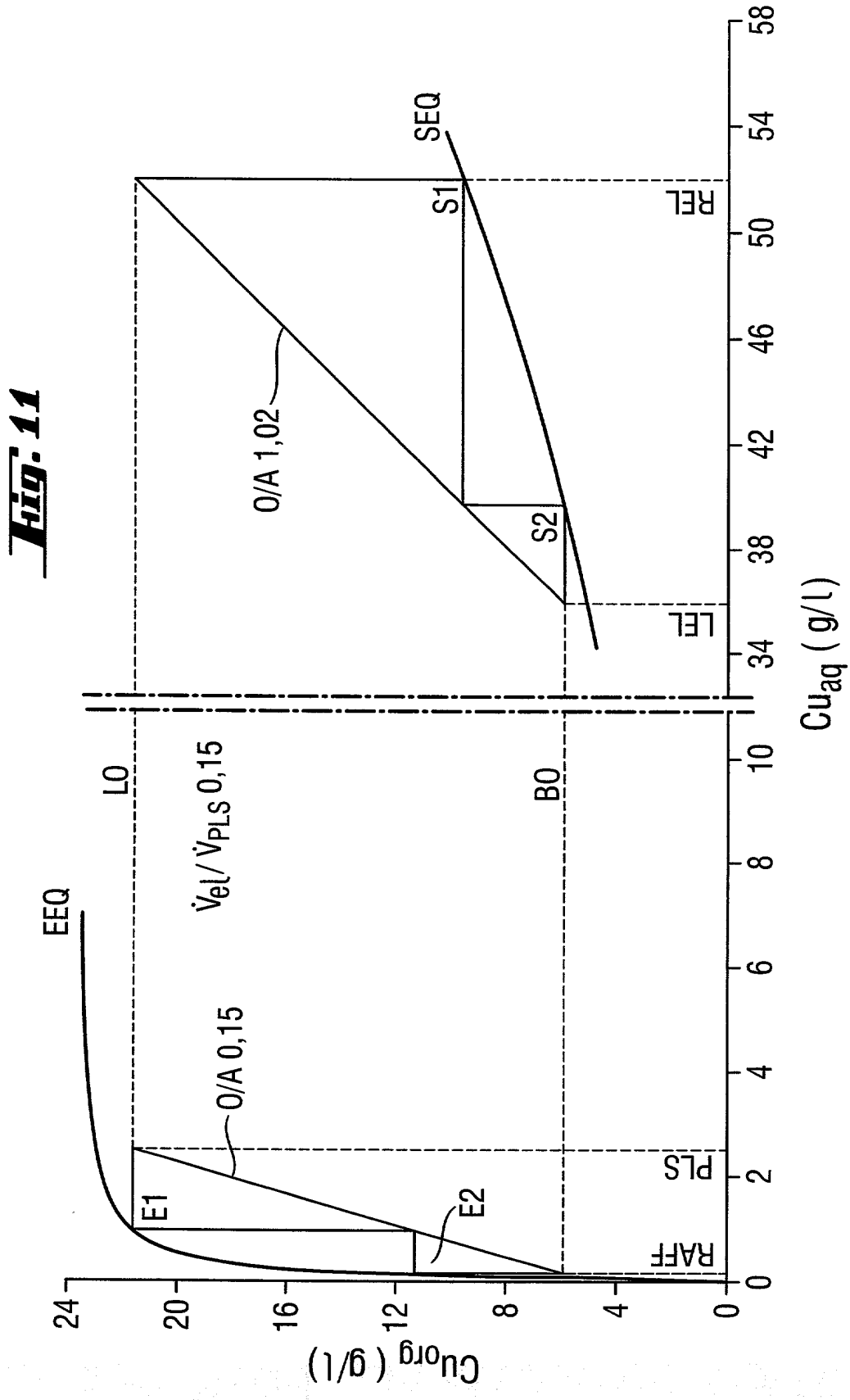
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Fig. 11

10/019967

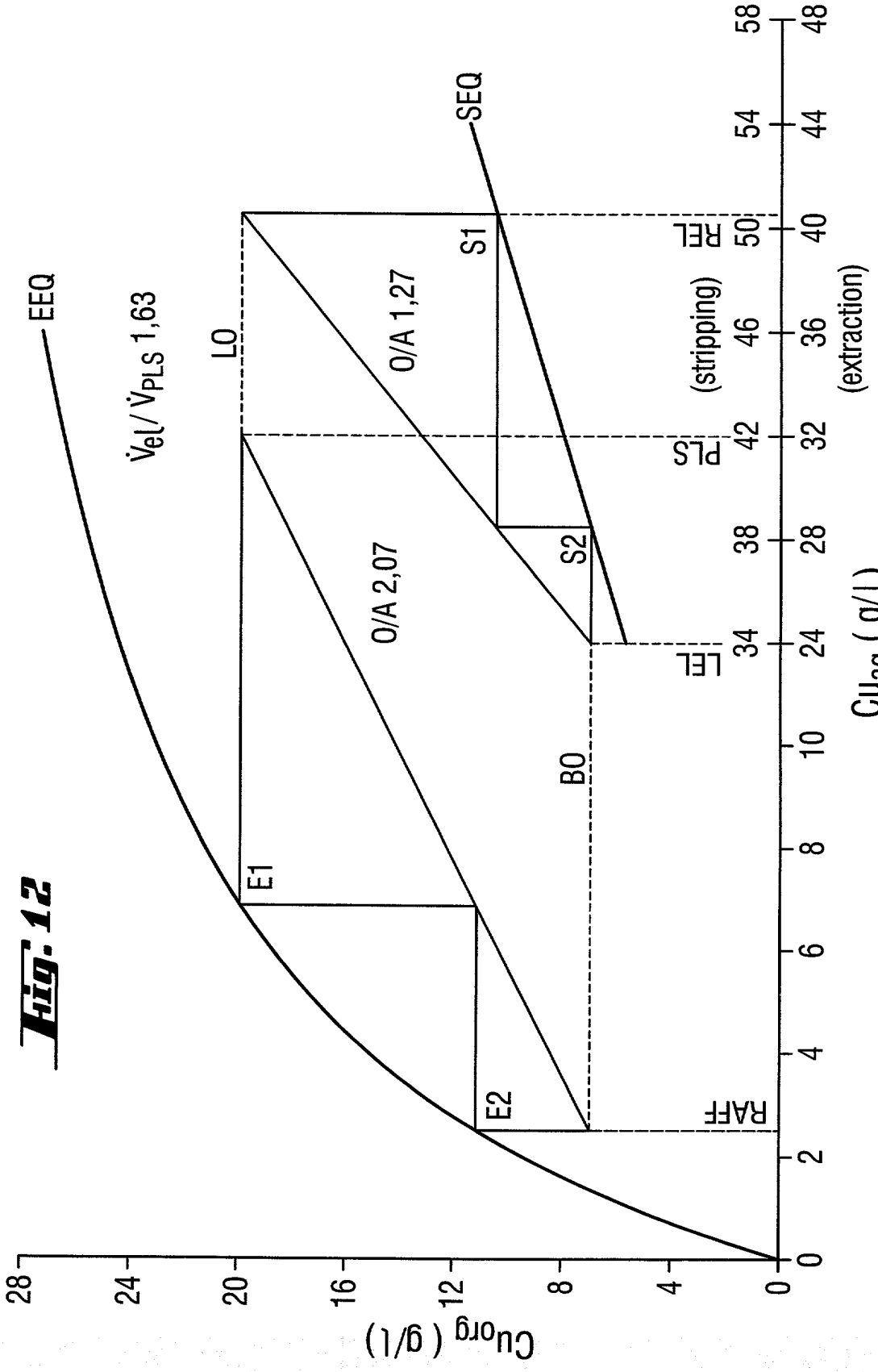


**Fig. 12**

99111 US

Docket No. _____

COMBINED DECLARATION AND POWER OF ATTORNEY FOR
ORIGINAL, DESIGN, NATIONAL STAGE OF PCT, SUPPLEMENTAL
DIVISIONAL, CONTINUATION OR CONTINUATION-IN-PART APPLICATION

As a below name inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled: A method for extracting copper from an aqueous solution

the specification of which

a. [ ] is attached hereto

b. [ ] was filed on _____ as application Serial No. _____ and was amended on _____. (if applicable).

PCT FILED APPLICATION ENTERING NATIONAL STATE

c. [X] was described and claimed in International Application No. PCT/FI00/00397 filed on 4 May 2000 and as amended on _____. (if any).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, § 1.56(a).

I hereby specify the following as the correspondence address to which all communications about this application are to be directed:

SEND CORRESPONDENCE TO: MORGAN & FINNEGAN, L.L.P
345 Park Avenue
New York, N.Y. 10154

DIRECT TELEPHONE CALLS TO:
(212) 758-4800

[ ] I hereby claim foreign priority benefits under Title 35, United States Code § 119(a)-(d) or under § 365(b) of any foreign application(s) for patent or inventor's certificate or under § 365(a) of any PCT international application(s) designating at least one country other than the U.S. listed below and also have identified below such foreign application(s) for patent or inventor's certificate or such PCT international application(s) filed by me on the same subject matter having a filing date within twelve (12) months before that of the application on which priority is claimed:

[ ] The attached 35 U.S.C. § 119 claim for priority for the application(s) listed below forms a part of this declaration.

| Country/PCT | Application Number | Date of filing (day, month, yr) | Date of Issue (day, month, yr) | Priority Claimed |
|---|---|---|---|---|
| Finland | 991111 | 14 May 1999 | | [X] YES [ ] NO |
| | | | | [ ] YES [ ] NO |
| | | | | [ ] YES [ ] NO |

[ ] I hereby claim the benefit under 35 U.S.C. § 119(e) of any U.S. provisional application(s) listed below.

| Provisional Application No. | Date of Filing (day, month, yr) |
|---|---|
| | |
| | |

ADDITIONAL STATEMENTS FOR DIVISIONAL, CONTINUATION OR CONTINUATION-IN-PART OR PCT INTERNATIONAL APPLICATION(S) (DESIGNATING THE U.S.)

I hereby claim the benefit under Title 35, United States Code § 120 of any United States application(s) or under § 365(c) of any PCT international application(s) designating the U.S. listed below.

| US/PCT Application Serial No. | Filing Date | Status (patented, pending, abandoned)/ U.S. application no. assigned (For PCT) |
|---|---|---|
| | | |
| US/PCT Application Serial No. | Filing Date | Status (patented, pending, abandoned)/ U.S. application no. assigned (For PCT) |
| | | |

[ ] In this continuation-in-part application, insofar as the subject matter of any of the claims of this application is not disclosed in the above listed prior United States or PCT international application(s) in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, § 1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or Imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

I hereby appoint the following attorneys and/or agents with full power of substitution and revocation, to prosecute this application, to receive the patent, and to transact all business in the Patent and Trademark Office connected therewith: John A. Diaz (Reg. No. 19,550), John C. Vassil (Reg. No. 19,098), Alfred P. Ewert (Reg. No. 19,887), David H. Pfeffer (Reg. No. 19,825), Harry C. Marcus (Reg. No. 22,390), Robert E. Paulson (Reg. No. 21,046), Stephen R. Smith (Reg. No. 22,615), Kurt E. Richter (Reg. No. 24,052), J. Robert Dailey (Reg. No. 27,434), Eugene Moroz (Reg. No. 25,237), John F. Sweeney (Reg. No. 27,471), Arnold I. Rady (Reg. No. 26,601), Christopher A.

Hughes (Reg. No. 26,914), William S. Feiler (Reg. No. 26,728), Joseph A. Calvaruso (Reg. No. 28,287), James W. Gould (Reg. No. 28,859), Richard C. Komson (Reg. No. 27,913), Israel Blum (Reg. No. 26,710), Bartholomew Verdirame (Reg. No. 28,483), Maria C.H. Lin (reg. No. 29,323), Joseph A. DeGirolamo (Reg. No. 28,595), Michael A. Nicodema (Reg. No. 33,199), Michael P. Dougherty (Reg. No. 32,730), Seth J. Atlas (Reg. No. 32,454), Andrew M. Riddles (Reg. No. 31,657), Bruce D. DeRenzi (Reg. No. 33,676), Michael M. Murray (Reg. No. 32,537), Mark J. Abate (Reg. No. 32,527), Alfred L. Haffner, Jr. (Reg. No. 18,919), Harold Haidt (Reg. No. 17,509), John T. Gallagher (Reg. No. 35,516), Steven F. Meyer (Reg. No. 35,613) and Kenneth H. Sonnenfeld (Reg. No. 33,285) of Morgan & Finnegan, L.L.P. whose address is: 345 Park Avenue, New York, New York, 10154; and Edward A. Pennington (Reg. No. 32,588), Michael S. Marcus (Reg. No. 31,727) and John E. Hoel (Reg. No. 26,279) of Morgan & Finnegan, L.L.P., whose address is 1775 Eye Street, Suite 400, Washington, D.C. 20006.

[x] I hereby authorize the U.S. attorneys and/or agents named hereinabove to accept and follow instructions from Outokumpu Oyj, Espoo, Finland as to any action to be taken in the U.S. Patent and Trademark Office regarding this application without direct communication between the U.S. attorneys and/or agents and me. In the event of a change in the person(s) from whom instructions may be taken I will so notify the U.S. attorneys and/or agents hereinabove.

Full name of sole or first inventor NYMAN, Bror

Inventor's signature* X _____ 26 October 2001 date

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Full name of second joint inventor, if any HULTHOLM, Stig-Erik

Inventor's signature* X _____ 26 October 2001 date

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[x] ATTACHED IS ADDED PAGE TO COMBINED DECLARATION AND POWER OF ATTORNEY FOR SIGNATURE BY THIRD AND SUBSEQUENT INVENTORS FORM.

* Before signing this declaration, each person signing must:

1. Review the declaration and verify the correctness of all information therein; and

2. Review the specification and the claims, including any amendments made to the claims.

After the declaration is signed, the specification and claims are not to be altered.

To the inventor(s):

The following are cited in or pertinent to the declaration attached to the accompanying application:

Title 37, Code of Federal Regulation, § 1.56

Duty to disclose information material to patentability.

(a) A patent by its very nature is affect with a public interest. The public interest is best served, and the most effective patent examination occurs when, at the time an application is being examined, the Office is aware of and evaluates the teachings of all information material to patentability. Each individual associated with the filing and prosecution of a patent application has a duty of candor and good faith in dealing with the Office, which includes a duty to disclose to the Office all information known to that individual to be material to patentability as defined in this section. The duty to disclose information exists with respect to each pending claim until the claim is canceled or withdrawn from consideration, or the application becomes abandoned. Information material to the patentability of a claim that is canceled or withdrawn from consideration need not be submitted if the information is not material to the patentability of any claim remaining under consideration in the application. There is no duty to submit information which is not material to the patentability of any existing claim. The duty to disclose all information known to be material to patentability is deemed to be satisfied if all information known to be material to patentability of any claim issued in patent was cited by the Office or submitted to the Office in the manner prescribed by §§1.97(b)-(d) and 1.98. However, no patent will be granted on an application in connection with which fraud on the Office was practiced or attempted or the duty of disclosure was violated through bad faith or intentional misconduct. The Office encourages applicants to carefully examine:

(1) prior art cited in search reports of a foreign patent office in a counterpart application, and

(2) the closest information over which individuals associated with the filing or prosecution of a patent application believe any pending claim patentably defines, to make sure that any material information contained therein is disclosed to the Office.

Title 35, U.S. Code § 101

Inventions patentable

Whoever invents or discovers any new and useful process, machine, manufacture, or composition of matter, or any new and useful improvement thereof, may obtain a patent therefor, subject to the conditions and requirements of this title.

Title 35 U.S. Code § 102

Conditions for patentability; novelty and loss of right to patent

A person shall be entitled to a patent unless –

(a) the invention was known or used by others in this country, or patented or described in a printed publication in this or a foreign country, before the invention thereof by the applicant for patent,

(b) the invention was patented or described in a printed publication in this or foreign country or in public use or on sale in this country, more than one year prior to the date of application for patent in the United States, or

(c) he has abandoned the invention, or

(d) the invention was first patented or caused to be patented, or was the subject of an inventor's certificate, by the applicant or his legal representatives or assigns in a foreign country prior to the date of the application for patent in this country on an application for patent or inventor's certificate field more than twelve months before the filing of the application in the United States, or

(e) the invention was described in a patent granted on an application for patent by another filed in the United States before the invention thereof by the applicant for patent, or on an international application by another who has fulfilled the requirements of paragraphs (1), (2), and (4) of section 371(c) of this title before the invention thereof by the applicant for patent, or

(f) he did not himself invent the subject matter sought to be patented, or

(g) before the applicant's invention thereof the invention was made in this country by another had not abandoned, suppressed, or concealed it. In determining priority of invention there shall be considered not only the respective dates of conception and reduction to practice of the invention, but also the reasonable diligence of one who was first to conceive and last to reduce to practice, from a time prior to conception by the other ...

Title 35, U.S. Code § 103

Conditions for patentability; non-obvious subject matter

A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said matter pertains. Patentability shall not be negatived by the manner in which the invention was made.

Subject matter developed by another person, which qualifies as prior art only under subsection (f) or (g) of section 102 of this title, shall not preclude patentability under this section where the subject matter and the claimed invention were, at the time the invention was made, owned by the same person or subject to an obligation of assignment to the same person.

Title 35, U.S. Code § 112 (in part)

Specification

The specification shall contain a written description of the invention, and of the manner and process of making and using it, in such full, clear, concise and exact terms also enable any person skilled in the art to which it pertains, or with which it is mostly nearly connected, to make and use the same, and shall set forth the best mode contemplated by the inventor of carrying out his invention.

Title 35, U.S. Code § 119

Benefit of earlier filing date in foreign country; right of priority

An application for patent for an invention filed in this country by any person who has, or whose legal representatives or assigns have, previously regularly filed an application for a patent for the same invention in a foreign country which affords similar privileges in the case of applications filed in the United States or to citizens of the United States, shall have the same effect as the same application would have if filed in this country on the date on which the application for patent for the same invention was first filed in such foreign country, if the application in

this country is filed within twelve months from the earliest date on which such foreign application was filed; but no patent shall be granted on any application for patent for an invention which had been patented or described in a printed publication in any country more than one year before the date of he actual filing of the application in this country, or which had been in public use or on sale in this country more than one year prior to such filing.

Title 35, U.S. Code § 120

Benefit or earlier filing date in the United States

An application for patent for an invention disclosed in the manner provided by the first paragraph of section 112 of this title in an application previously filed in the United States, or as provided by section 363 of this title, which is filed by an inventor or inventors named in the previously filed application shall have the same effect, as to such invention, as though filed on the date of the prior application, if filed before the patenting or abandonment of or termination of proceedings on the first application or an application similarly entitled to the benefit of the filing date of the first application and if it contains or is amended to contain a specific reference to the earlier filed application.

Please read carefully before signing the Declaration attached to the accompanying Application.

If you have any questions, please contact Morgan & Finnegan, L.L.P.

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Rev. 5/21/98

PATENT
Docket No. _____

ADDED PAGE TO COMBINED DECLARATION
AND POWER OF ATTORNEY FOR
SIGNATURE BY THIRD AND SUBSEQUENT INVENTOR

300 Full name of third joint inventor, if any LILJA, Launo

Inventor's signature* X _____ 26 October 2001 date

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400 Full name of fourth joint inventor, if any LINDELL, Esa

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500 Full name of fifth joint inventor, if any EKMAN, Eero

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X) Attached an added page

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PATENT
Docket No. _____

ADDED PAGE TO COMBINED DECLARATION
AND POWER OF ATTORNEY FOR
SIGNATURE BY THIRD AND SUBSEQUENT INVENTOR

600 Full name of ~~third~~ sixth joint inventor, if any LYYRA, Juhani

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700 Full name of ~~fourth~~ seventh joint inventor, if any KUUSISTO, Raimo

Inventor's signature* X _____ 26 October 2001
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800 Full name of ~~fifth~~ eight joint inventor, if any PEKKALA; Pertti

Inventor's signature* X _____ 26 October 2001
date

Residence ~~Vehaksentie 45 B~~

Citizenship ~~Finnish~~

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